Metrological investigation of nanostructured polymer surfaces replication using atomic force microscopy

Uncertainty evaluation in the surface replication fidelity assessment of moulded specimens at the 100 nm scale

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Replication assessment of nanostructured polymer surfaces using atomic force microscopy

Uncertainty evaluation in the surface replication fidelity assessment of moulded specimens at the 100 nm scale

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Abstract

Polymer specimens have been manufactured by injection moulding and measured by atomic force microscopy (AFM) with the aim to investigate the possibility of replicating their surfaces with good fidelity at the sub-µm dimensional scale. Three different cases with surface features in the 100 nm amplitude range on the surface have been analysed: specimens with random and periodic surface examined in the same production batch and specimens with periodic surface produced in two different batches. The assessment of the AFM measurement uncertainty and its use in the replication analysis is discussed. Results show that high replication fidelity of the polymer specimens can be achieved in all the cases examined.

1. Introduction

Product miniaturisation and micro-systems have been strong drivers of a technological change, with a significant impact on the manufacturing industry. Precision moulding and micro injection moulding (µIM) are the key moulding technologies for polymer micro-parts and parts with micro/nano surfaces manufacture.

The miniaturisation of moulded parts and features leads to new challenges in injection moulding processes. Specially developed solutions are needed in all production steps [1].

One of the key challenges in advanced µIM technology is the achievement of a full surface replication of the tool insert component when moulding the polymer melt. This aspect is particularly critical when dealing with increasingly small dimensional scales in micro- and nano-structured surfaces.

Because of the replication nature of moulding processes, the accuracy needed for micro moulded components manufacture must be ensured by means of a metrological approach to surface replication and dimensional control of both tools and replicated parts ([2], [3] and [4]).

In this context, metrology has an extremely important role to play. Advanced product concepts are based on integrated processes and process chains include different materials and span across different dimensional scales. These characteristics require detailed knowledge of not only absolute dimensions and geometrical quantities, but also about the uncertainty of measurement, because this is a decisive parameter when dealing with quality control of micro manufactured components [5].

2. Sub-micro structured polymer surfaces manufacture

The polymer surfaces used in the investigation have been replicated by injection moulding using a commercially available acrylonitrile butadiene styrene (ABS) Cycolac KJY 039075 produced by Borg Warner with a grey colour. Two nickel roughness standards, manufactured by Rubert & Co Ltd., UK, have been used as tool inserts in mould mounted on a conventional injection moulding machine (Ferromatik Milacron K60) with a reciprocating screw of 35 mm in diameter and a clamping force of 60 kN.

The types of standards used are listed in Table 1 and their characteristics are specified according to [6]. An example of roughness standard tool insert and of a polymer specimen replicated by injection moulding can be seen in Figure 1.

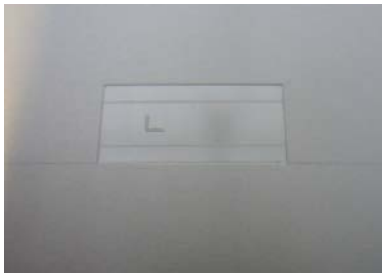
Table 1: Nominal characteristics of the nickel standards used as masters for the production of the injection moulded parts.

Master number	ISO type	Nominal roughness parameter values	Shape
503	D	*Ra* = 0.1 µm	4 × 1.25 mm random
529	C	*RSm* = 10 µm *Pt* = 0.3 µm *Ra* = 0.1 µm	Sine wave

Table 2: Injection moulding parameters used for the production of the polymer parts; (*): including packing, cooling and demoulding phases.

Melt temperature /°C	230
Mould temperature /°C	50
Injection speed /mm s^{-1}	50
Max injection pressure /bar	41
Total cycle time /s (*)	≈ 60

(a)



(b)

Figure 1: Examples of roughness standard tool insert (a) and replicated specimen (b).

3. Process characterization by areal surface AFM measurements

3.1 Process repeatability and replication fidelity within the same production batch

In order to investigate the replication fidelity of the injection moulding process, 18 replicated polymer specimens for both reference standards 503 and 529 have been selected in the same production batch and measured using an atomic force microscope (AFM).

The measured surfaces (*Sa* roughness parameter defined in [7] was evaluated), acquired in reproducibility conditions, have been compared with the measurement results of the reference standards (tool inserts), also obtained using an atomic force microscope (AFM), considering their respective uncertainty intervals (see § 4 below for further details on uncertainty evaluation).

The measurements have been acquired in the same area on each specimen which was identified using a mark on the surface of the tool inserts (reference roughness standards) consequently transferred on the surface of the replicated specimens. Furthermore, the same area of 250 µm × 250 µm was analysed. This was ensured because, especially when comparing different measurements (that is, in this case the same area in the nickel tool replicated in different components, i.e., the moulded parts), it is essential that the spatial bandwidths match. A simple, but effective way to achieve this is by measuring the same profile length or area with the same amount of points [8]. Finally, a 2-D Gaussian filter according to [9] (S-filter at 2.5 µm) was applied. Results are summarised in Figure 2.

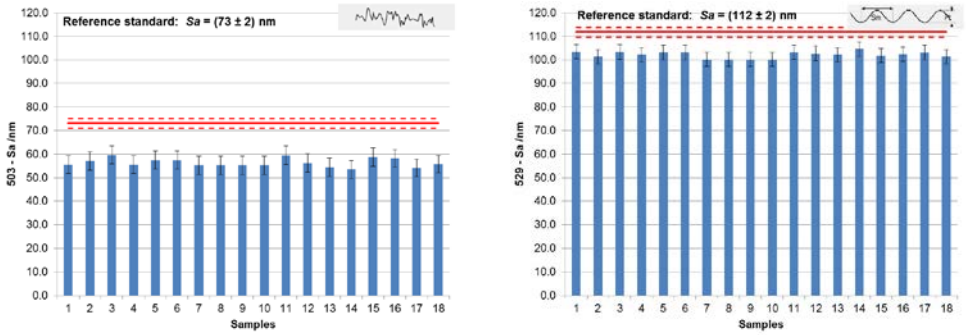


Figure 2: AFM measurements results (columns) of two series of replicated samples from a single batch production. Results are given for *Sa* parameter. The bars represent the expanded uncertainty evaluated. AFM results (solid red lines) of reference roughness standards are also in the graphs together with the expanded uncertainty intervals (dashed red lines).

3.2 Process reproducibility and replication fidelity in two different production batches

Two different batches of production have been measured using an AFM, considering six samples for each batch, i.e., twelve replicated specimens for the selected 529 specimen surface.

The measured surfaces (*Sa* roughness parameters defined in [7] were evaluated) have been compared with the AFM measurement results of the reference standards (tool inserts), considering their respective uncertainty intervals (see § 4 below for further details on uncertainty evaluation) and, also, the measurement procedure presented in § 3. Results are summarised in Figure 3.

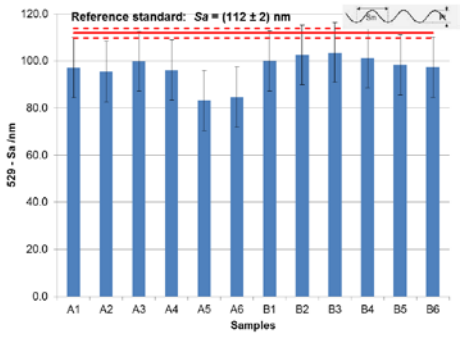


Figure 3: AFM measurements results (columns) of the periodic series of replicated samples (529) from a two batches production. Results are given for the *Sa* parameter. The bars represent the expanded uncertainty evaluated. AFM results (solid red lines) of reference roughness standards are also in the graphs together with the expanded uncertainty intervals (dashed red lines).

4. Uncertainty model

Results of measurements operations need always to be specified within their uncertainty intervals. If the measurement approach/instrument results in a large measurement uncertainty, then no clear conclusion with respect to compliances can be made.

In this study, a measurement result *y* and its expanded uncertainty *U* have been considered as $y \pm U$, where *U* is determined with a coverage factor $k = 2$, i.e., considering an approximated expanded interval of 95%.

The expanded uncertainty *U* was evaluated according to [10], in which the so-called "substitution method", normally adopted for coordinate measuring machines measurements, was adapted for measurements from AFM. The uncertainty evaluation flow and the considered contributors are shown in the Figure 4. In Table 3 an example of uncertainty budget of measurements associated with the two production batches of 529 replicated specimens is shown.

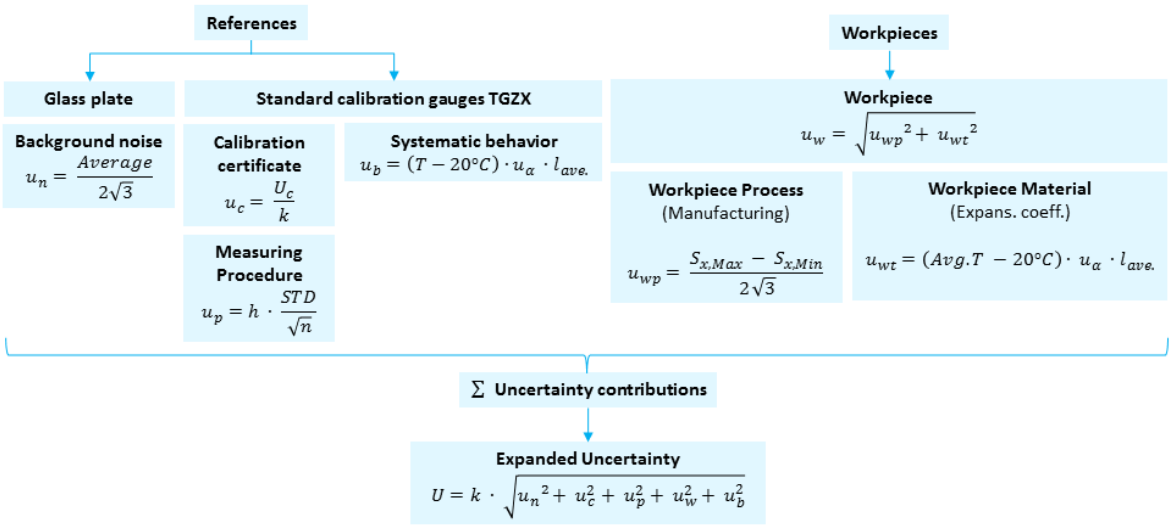


Figure 4: Uncertainty model used in the investigation.

Table 3: Uncertainty contributors of AFM measurements on two-batches 529 polymer specimens.

Uncertainty contributors					Expanded combined uncertainty ($k = 2$)
u_n /nm	u_c /nm	u_p /nm	u_w /nm	u_b /nm	U /nm
0.1	0.7	0.3	6.3	<0.001	13

5. Discussion

A good replication of the polymer specimens can be deduced by the analysis for all cases considered. Both random and periodic surfaces, analysed in the same batch, even though they do not reach exactly the same amplitude (*Sa* parameter) of the references (Figure 2), have congruent intervals of uncertainty. Moreover, the average difference between the *Sa* values of the polymer samples and the references averages ($Sa_{poly} - Sa_{ref}^{ave}$) is very close to the evaluated uncertainty.

When two different batches are considered, similar results can be observed (see Figure 3): congruent intervals of uncertainty and small distance from the reference. The uncertainty contributor u_w (see Table 3), however, reveals a larger variability of the process.

Furthermore, a good process capability can be also deduced for random and periodic surfaces, both in the same batch and in different batches, from the maximum deviation among the measured surfaces ($Sa_{poly}^{\max} - Sa_{poly}^{\min}$). This quantity is again well represented by the uncertainty evaluated for the replicated surfaces (same order of magnitude).

6. Conclusion

In this paper an investigation of the replication fidelity of different moulded specimens has been presented. Three different cases have been analysed: samples with random and periodic surface examined in the same batch and samples with periodic surface examined in two different batches. Results show that a good amplitude replication can be achieved for all the cases with surface amplitude in the 100 nm dimensional range.

The use of the uncertainty in the replication analysis has also been shown. Indeed, the uncertainty has two main contributors: one is related to the variability of the replicated surface (different samples considered) and the other one is related to the measuring instrument (repeated measurements are necessary to well describe this variability). Hence, it is extremely important to state the evaluation of the uncertainty in a proper way so that it can be a useful tool in the investigation of the replication. If the uncertainty associated with the instrument becomes too high, it would hide the variability of the surfaces and of the manufacturing process and hence the replication would not be observed.

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